Carderock Division
Naval Surface Warfare Center
West Bethesda, MD 20817-5700

NSWCCD-TR–2000/003+CR 20 November 1999

Materials, Structures and Survivablility Directorate

Technical Report

High Thermal Conductivity Composite Structures

by

John Bootle

XC Associates, Inc

Berlin, New York

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	20 November 1999	Final May - Nov 1999

4. TITLE AND SUBTITLE
High Thermal Conductivity Composite Structures

5. FUNDING NUMBERS
Contract number N00167-99-C-0047

6. AUTHOR(S)
John Bootle

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
XC ASSOCIATES, INC
28 SOUTH MAIN STREET
BERLIN, NY 12022

8. PERFORMING ORGANIZATION REPORT NUMBER
1000-0282

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
NAVAL SURFACE WARFARE CENTER
CARDEROCK DIVISION (CODE 681)
9500 MACARTHUR BOULEVARD
WEST BETHESDA, MD 20817-5700

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NSWCCD-TR–2000/003+CR

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

The work accomplished in this SBIR project demonstrated that the addition of boron nitride (BN) powder to a composite laminate significantly increases the through the thickness thermal conductivity (Kz) of a composite laminate. The importance of this work is that the improved Kz results in significantly lower operating temperatures for thermal applications such as composite thermal planes for advanced electronic applications and space based radiators. The advantage of this material compared to competing materials that it can be used to fabricate high strength, high thermal conductivity, relatively thin structures less than 0.050-inch thick.

Typical graphite fiber reinforced composite thermal planes have an in-plane thermal conductivity (Kx and Ky) in the range 300-650 w/m/K, based on the fiber selection. But, the relatively low Kz of a typical composite laminate significantly reduces the efficiency of the thermal plane due to the high impedance of getting heat in or out of the laminate. Finite element analysis of typical composite thermal planes shows that by increasing the Kz from about 1 w/m/K for a typical laminate to about 4 w/m/K, as achieved in this project, results in temperature reductions in the order of 30%.

14. SUBJECT TERMS
High Thermal Conductivity Composite
Enhanced Kz
Lightweight Thermal Planes
Spacecraft Thermal Radiators
Thermal Management

15. NUMBER OF PAGES
20

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

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Administrative information

This final report of a Phase I Small Business Innovation Research (SBIR) program covers work conducted under contract N00167-99-C-0047, "High Thermal Conductivity Composite Structures" by XC Associates, Inc, Berlin, NY. The work demonstrated that a simple method of adding boron nitride powder increased the thermal conductivity of a composite laminate for thermal management applications. The thermal and mechanical properties of laminates were measured. This work has direct application for improved design of composite thermal planes for avionics and space applications.

Funding for the work was provided by the Ballistic Missile Defense Organization's SBIR program office. The micrograph project described in section 3.8, in addition to the SBIR scope of work, was funded directly by XC Associates.

Acknowledgements

XC Associates would like to thank Albert Bertram of the Naval Surface Warfare Center and Roger Gerzeski of the Air Force Research Laboratory for their support and guidance.

Professor Ron Bucinell of Union College carried out mechanical testing.

Steve Perrucci of Union College carried out the micrographs as part of his final year project.

1 Introduction

The work accomplished in this SBIR project demonstrated that the addition of boron nitride (BN) powder to a composite laminate significantly increases the through-the-thickness thermal conductivity (Kz) of a composite laminate. The importance of this work is that the improved Kz results in significantly lower operating temperatures for thermal applications such as composite thermal planes for advanced electronic applications and space based radiators. The advantage of this material compared to competing materials is that it can be used to fabricate high strength, high thermal conductivity, relatively thin structures less than 0.050-inch thick.

Typical graphite fiber reinforced composite thermal planes have an in-plane thermal conductivity (Kx and Ky) in the range 300-650 w/m/K, based on the fiber selection. But, the relatively low Kz of a typical composite laminate significantly reduces the efficiency of the thermal plane due to the high impedance to getting heat in or out of the laminate. Finite element analysis of typical composite thermal planes shows that by increasing the Kz from about 1 w/m/K for a typical laminate to about 4 w/m/K, as achieved in this project, results in temperature reductions in the order of 30%.

This project was carried out using Amoco (now BP Amoco) K-800X fiber, prepregged using Hexcel 954-3 resin. Since the resin, rather than the fiber, dominate Kz, the improvement in Kz is expected to be applicable to any carbon fiber laminate.

A number of companies have expressed interest in the work carried out during this project and XCA is working with them to commercialize the technology.

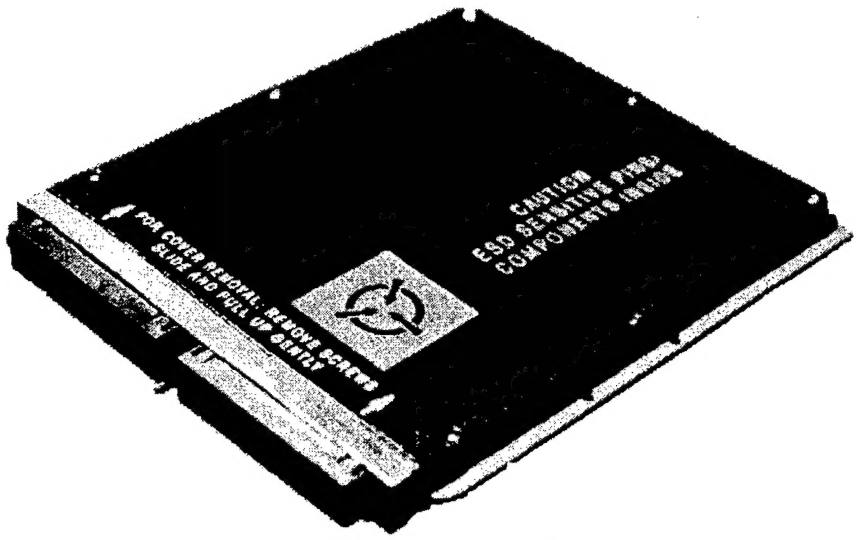

Figure 1. Photograph of typical composite thermal core and covers fabricated by XC Associates

2 Thermal results

2.1 Summary

The addition of boron nitride powder, BN, to a composite laminate increased the through-the-thickness thermal conductivity, Kz. However, the addition of the BN also reduced the fiber volume fraction. Hence, there was a decline in the in-plane thermal conductivity, Kx and Ky. Finite element analysis shows the net result was an improvement in overall thermal performance as discussed in paragraph 2.8.

Samples were cured using both a press cure without bleeding resin during the cure and an autoclave cure with significant resin bleed from the laminate during cure. The results showed that the Kz of the autoclave cure laminates was significantly higher than for the press cure. The reason for this requires further work, but a preliminary explanation is that the autoclave laminate has a higher fiber volume and the BN powder was more evenly distributed within the laminate.

2.2 Thermal conductivity Kz, press cured laminate, no resin bleed.

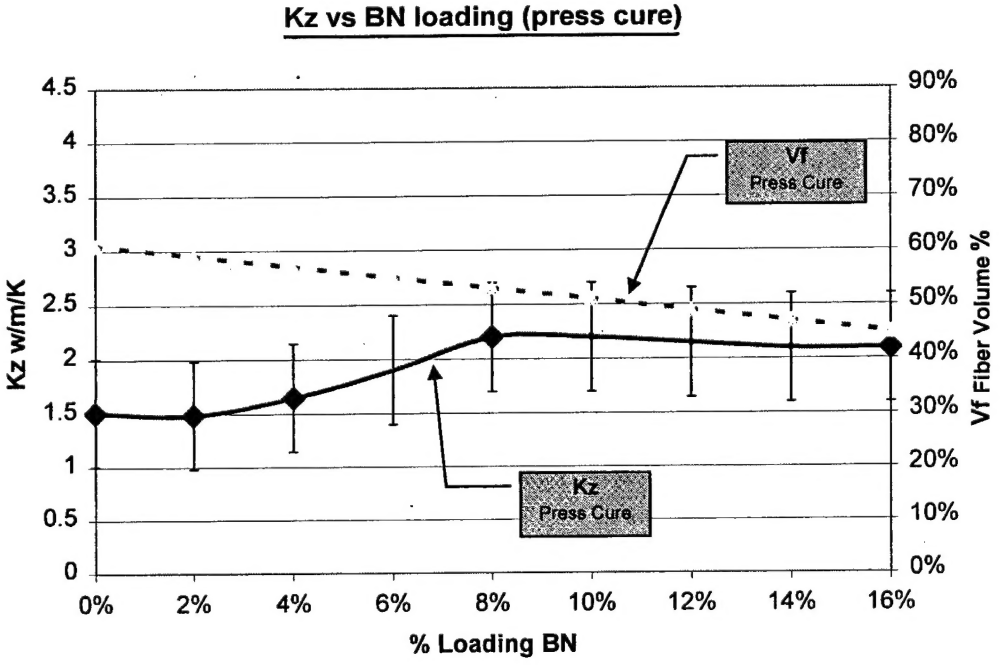

Figure 2. Kz for a press cured laminate

2.3 Thermal conductivity Kz, autoclave cure laminate, with resin bled.

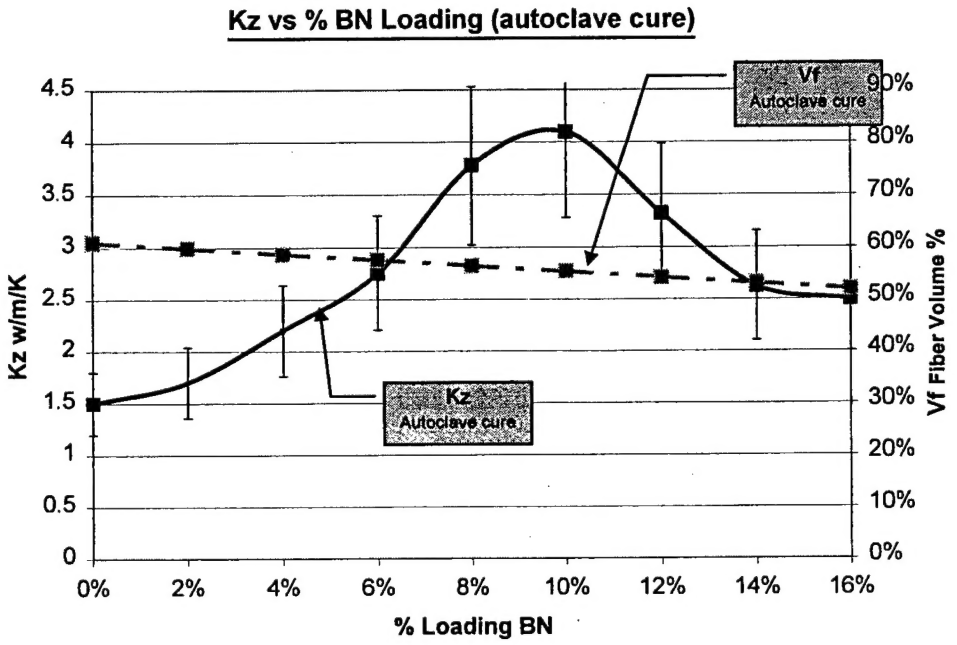

Figure 3. Kz for autoclave cured laminate

2.4 Graph comparing Kz for press and autoclave cured laminates

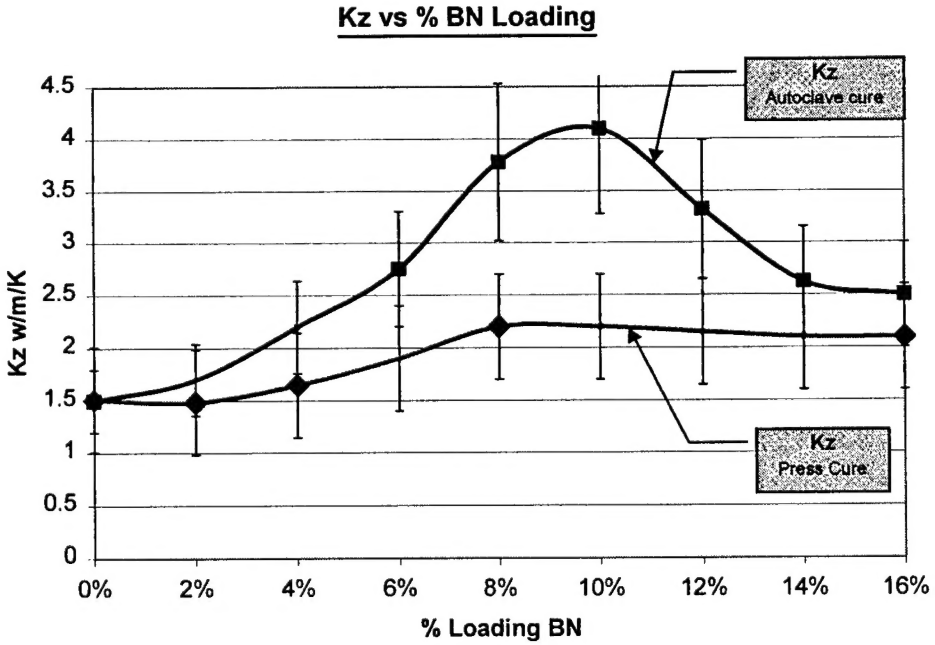

Figure 4. Kz for press and autoclave cured laminates

2.5 Description of graph

2.5.1 Laminate samples were cured using press cure during which there was very little resin bled from the laminate. During the autoclave cure cycle resin was bled from the laminate.

2.5.2 The fiber faction for the unfilled laminate was calculated from the prepreg data as presented in Table 3.

2.5.3 The fiber fraction of the filled laminates was calculated from the ratio-cured thickness of the filled laminate to the unfilled laminate after cure.

2.5.4 The laser flash method was used to measure the diffusivity, specific heat and density of the samples. The thermal conductivity K was calculated as Equation 1.

$$K = \alpha . \rho . Cp \qquad \textbf{Equation 1}$$

Where α = Thermal diffusivity

ρ = Density of laminate, 1.8 gr/cm^3 @25°C

Cp = specific heat of laminate, 0.85 J/gr. Deg @ 25°C

2.6 Verification of diffusivity and fiber fraction

The graph in Figure 5 shows the measured in-plane thermal conductivity, Kx, plotted against the theoretical value calculated from Equation 2. The good agreement gives high confidence to the laser flash method and the calculated Vf.

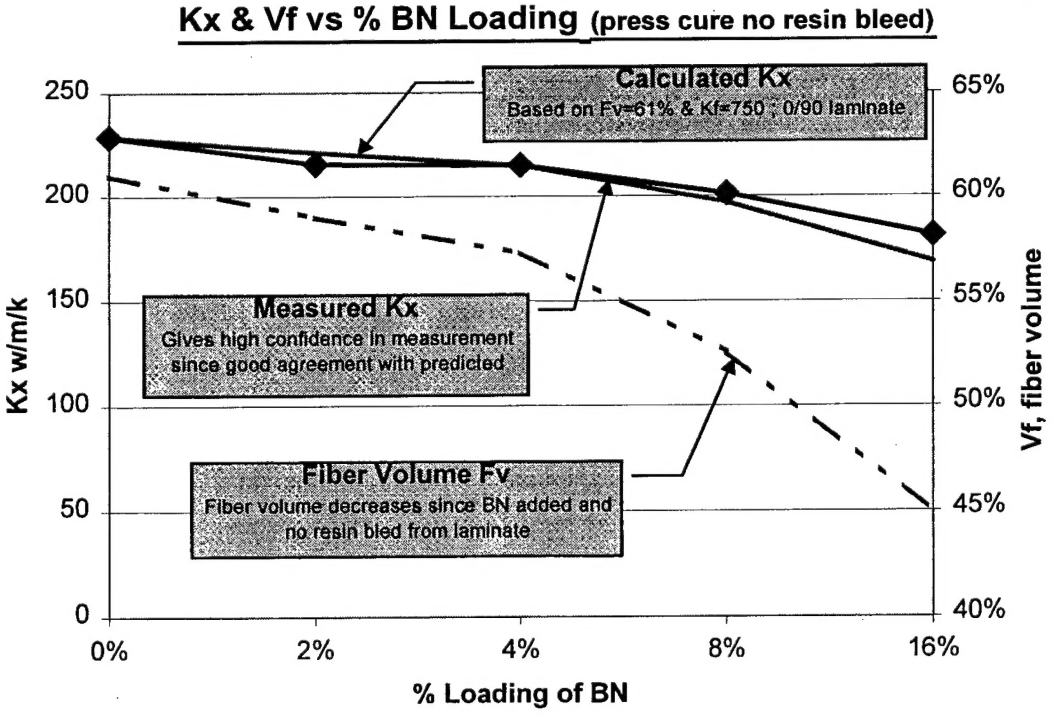

Figure 5. Kx and Vf vs % BN loading

2.6.1 Figure 5 also shows the variation of the measured Kx against the calculated fiber volume for the press-cured samples. (The laminate was a $0^o/90^o$ laminate so Kx=Ky). The fiber thermal conductivity of about 750 w/m/K was derived from data provided by Amoco.

2.7 Discussion of results

2.7.1 The laminates were fabricated by adding a predetermined weight of BN powder to each layer of the prepreg during the lay-up process. Since material was being added to the prepreg the volume increased and hence the fiber volume, Vf, decreased as more BN was added. This is important since in-plane thermal conductivity Kx and Ky is determined by the relationship

$$K_L = K_f.V_f + K_mV_m \approx K_f.V_f \qquad \textbf{Equation 2}$$

The thermal conductivity of the laminate is dominated by the fiber since $K_f >> K_m$
If the fibers are not orientated along the heat flow direction, the Equation is modified as

$$K_x = K_L.\sin^2\varphi \qquad K_y = K_L.\cos^2\varphi \qquad \textbf{Equation 3}$$

Where φ = angle of fibers of particular ply
K_f = Longitudinal thermal conductivity of fiber
K_m = Thermal conductivity of matrix
V_f = Fiber volume
V_m = matrix fiber volume

2.7.2 From Equations 2 and 3 it can be seen that the thermal conductivities, Kx and Ky are controlled by the fiber conductivity since this much greater than the resin conductivity.

2.7.3 There was considerable scatter in the results of Kz obtained from different autoclave cured samples. The reason is that the resin bleed varied among the samples indicating this is an important variable. More work needs to be carried out in this area.

2.7.4 Since the resin dominates Kz, it is expected that the improvement in Kz presented above will also be applicable to laminates fabricated from other fibers.

2.7.5 A preliminary investigation based on two samples showed that larger particle sizes resulted in less improvement in Kz. The effect of smaller particle sizes needs to be more completely investigated.

2.8 Practical importance of the results

Using the measured Kz results from Figure 3, and calculating the corresponding Kx and Ky (from Equations 2 and 3), the temperature of a typical SEM-E thermal plane was calculated. The results showed that the addition of 8%-10% BN to a laminate results in a 30% temperature reduction. This analysis assumed a typical SEM-E heatsink loaded with 65 watts of heat applied uniformly across the center of the heatsink while the rails were maintained at temperature T_0. A graph of the temperature difference ΔT is presented in Figure 6.

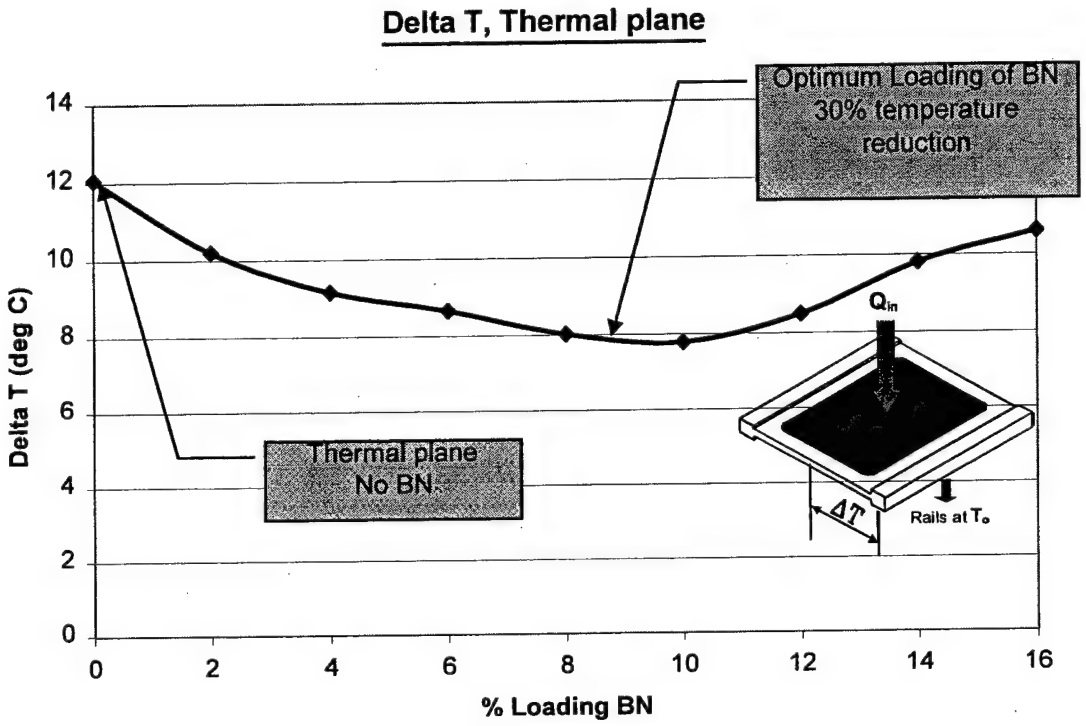

Figure 6. Improved performance of thermal core

3 Mechanical results

3.1 Summary of mechanical properties

Table 1. Comparison of mechanical properties

Property	Al-Be HIP'd AM162H	Aluminum 6061-T6	Composite K-800X / 954-3 0/90 laminate
Density lb/in^3	0.076	0.098	**0.065**
Thermal conductivity w/m/K	Kx = 210 Ky = 210 Kz = 210	Kx = 187 Ky = 187 Kz = 187	**Kx = 220** **Ky = 220** **Kz = 4**
Modulus of Elasticity Msi	E = 28 G n/a	E =10 G = 3.8	**Ex = 22** **Ey = 22** **G =4.7**
Tensile Strength ksi	Sx = Sy =28	Sx = Sy = 37	**Fx = 42** **Ky = 42**

3.1.1 The results presented in Table 1 are for a 0/90 laminate fabricated from K-800X/954-3 laminate filled with 8% BN.

3.1.2 Varying the fiber type and orientation, as shown in Equations 2 and 3 can optimize the values for thermal conductivities Kx and Ky. Typical values for Kx of 290- 530 w/m/K may be achieved using K13C2U, K-800X, or K-1100 respectively.

3.1.3 Stiffness and strength of the laminate are also determined by the fiber orientation.

3.1.4 The results of mechanical testing carried out on the press cure laminates are presented on the following pages.

3.2 Tensile modulus

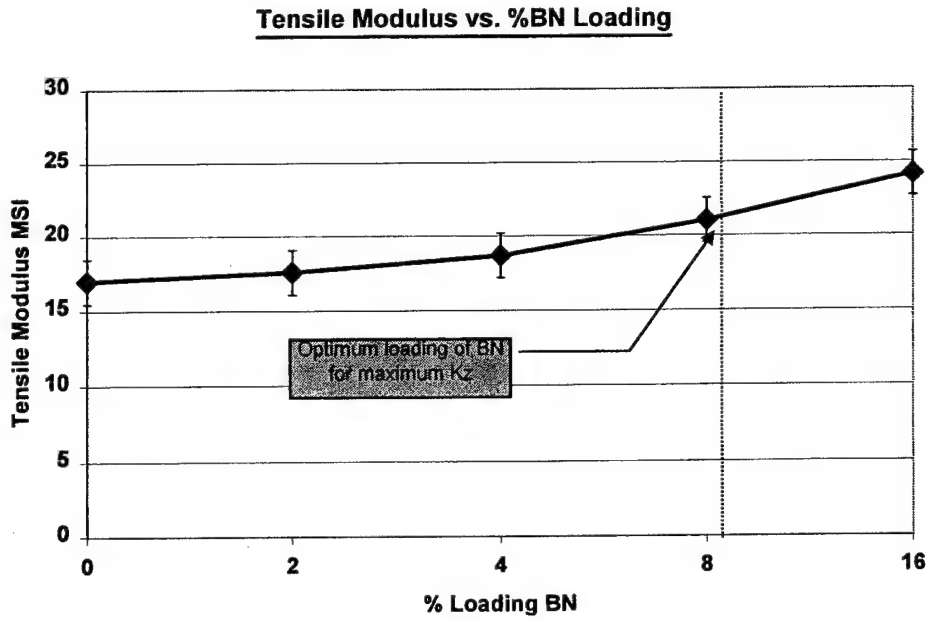

Figure 7. Tensile modulus

3.3 Tensile strength

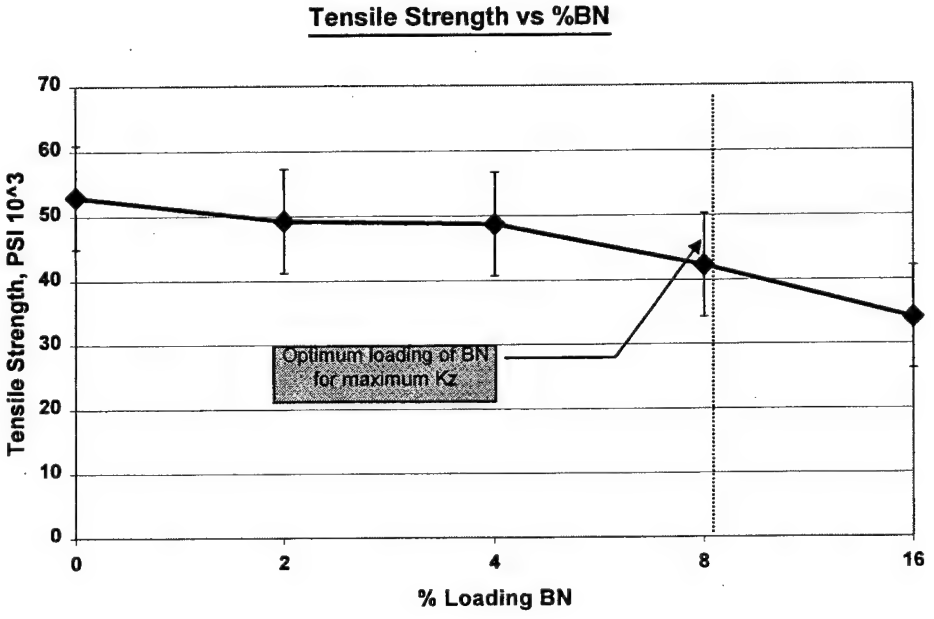

Figure 8. Tensile strength

3.4 Compression strength

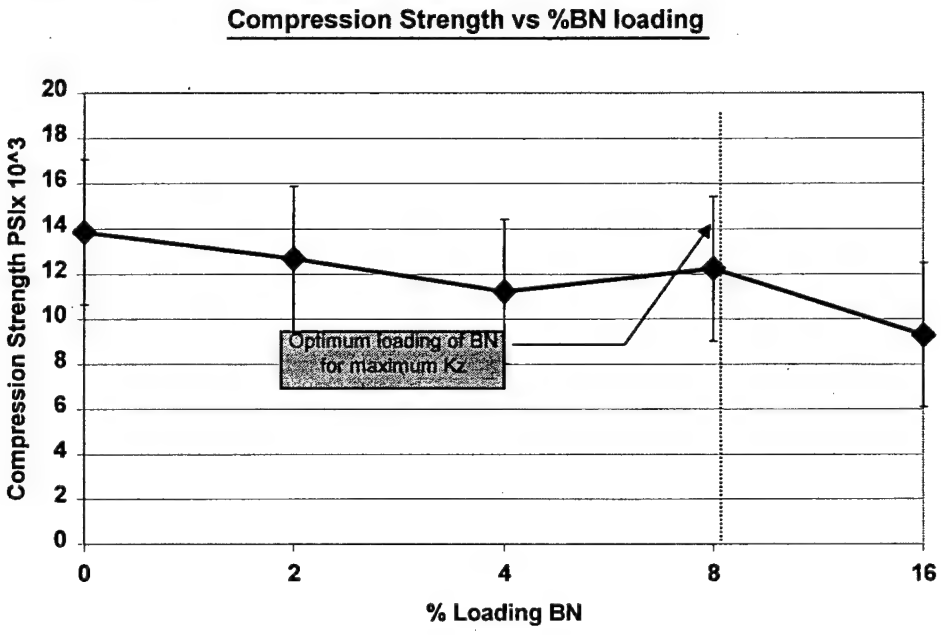

Figure 9. Compression strength

3.5 Shear strength

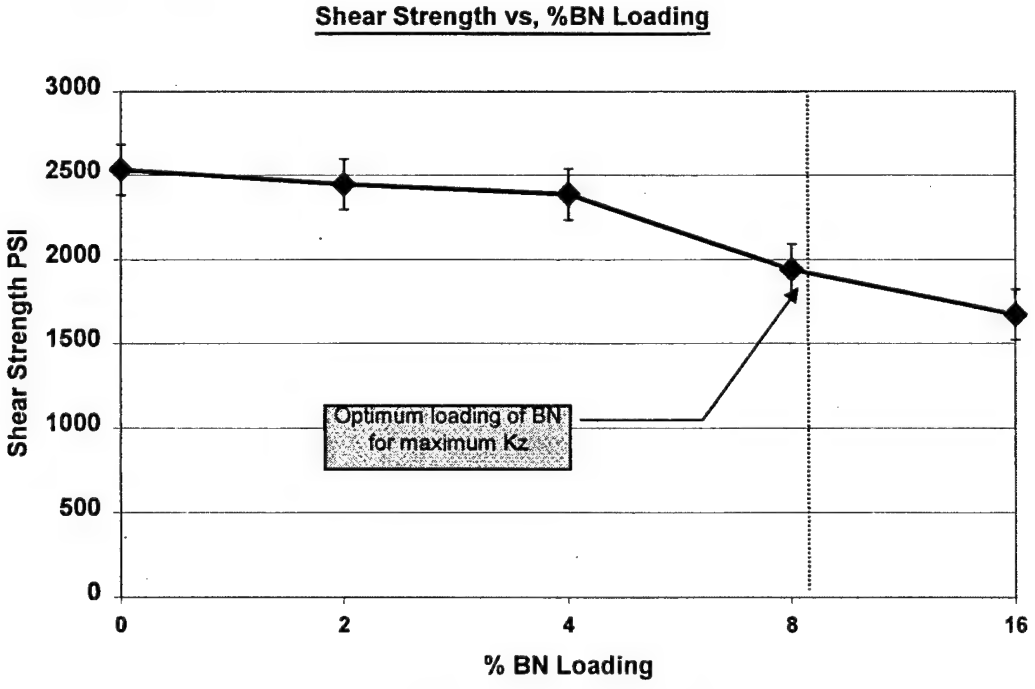

Figure 10. Shear strength

3.6 Discussion of mechanical results

3.6.1 The mechanical results indicate that the addition of BN powder reduces the laminate strength. This is expected since the addition of BN reduced the fiber volume percentage of the laminate. The measured strength of the laminate presented in Table 1 compares the strength of the laminate loaded with 8% BN compared to typical metals. Using the method of fabrication described in paragraph 5, Fabrication method, it is straightforward to only add BN to the areas with high thermal inputs and hence, achieve the highest structural strength.

3.6.2 We expect the mechanical strength to be related to the BN and resin volume percentages, therefore we would expect some variation between press and autoclave cure. This will be investigated in future work.

3.6.3 The tensile modulus increases with the addition of BN powder (see Figure 7. Tensile modulus). This result is contrary to our expectation but may be explained by the fact that the mixture of BN and resin has a higher stiffness than resin alone.

3.7 Mechanical test samples

3.7.1 The following tests were used to characterize the laminate; mechanical tests were only carried out on the press-cured samples.

Table 2. Material test samples

Test	ASTM	Comment
Tension	ASTM D 3039	5 test coupons from each sample
Short beam shear	ASTM D 2344	5 test coupons from each sample
Compression	ASTM D 3410	5 test coupons from each sample

3.7.2 The test samples were cut from a 12-inch x 12-inch x 0.1-inch thick laminate as shown in Figure 11.

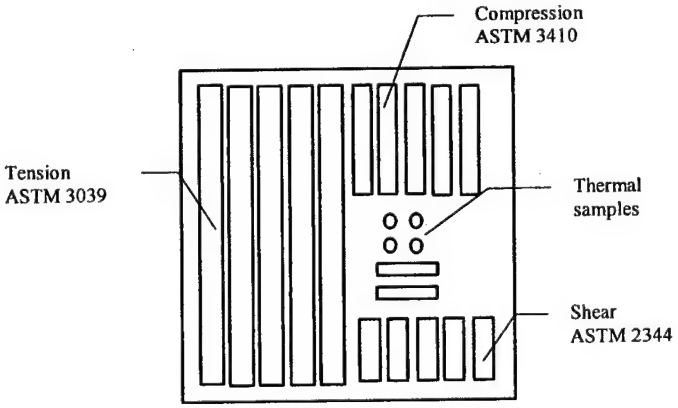

Figure 11. Layout of samples on 12-inch square test panel

3.8 Microscopic

3.8.1 The microscopic analysis of the samples is an on-going final year project being carried out at Union College, The objective of the work is to determine the distribution of the BN powder within the laminate. Data from these micrographs will be used in further work to develop a theoretical model of heat flow through the laminate.

3.8.2 The results presented here are for the press cured laminates; micrographs of the autoclave samples will be presented in a later report.

3.8.3 Samples were polished, then examined under a microscope to determine the location of the fibers, location of the BN, and the void content. The photographs of the micro sections are presented below.

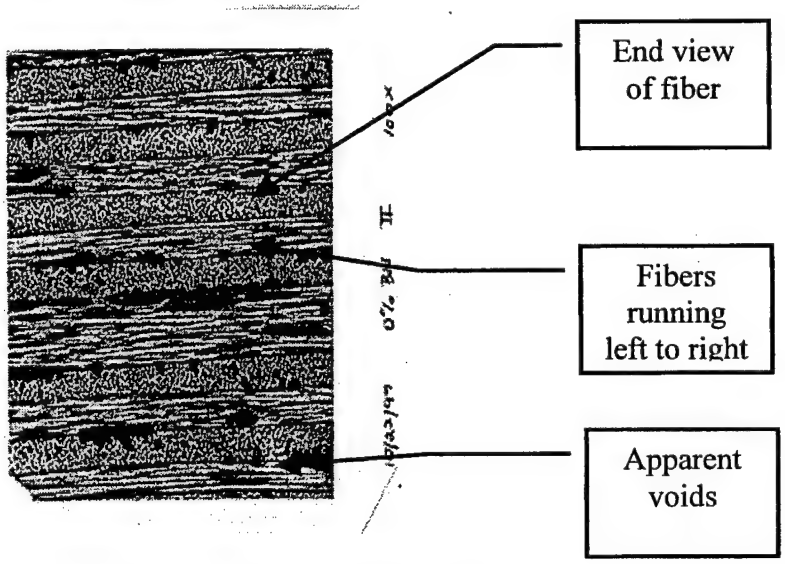

Figure 12. Sample showing 0% BN fill

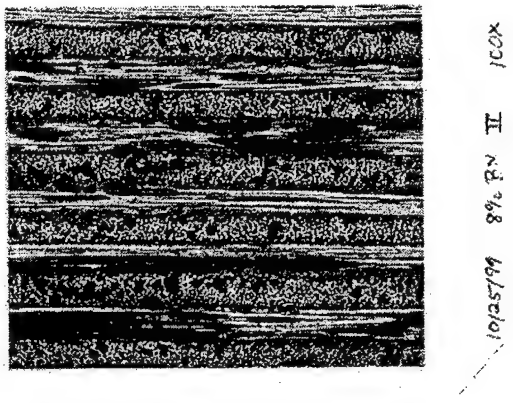

Figure 13. Sample showing 8% BN fill

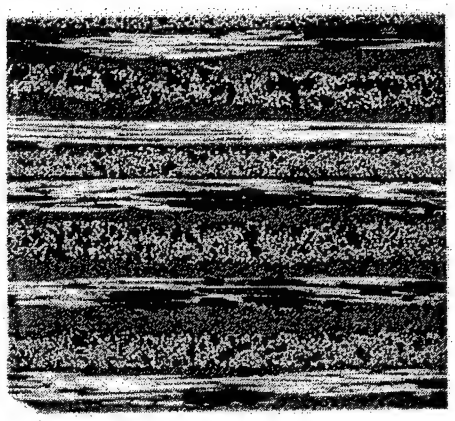

Figure 14. Sample showing 16% BN fill

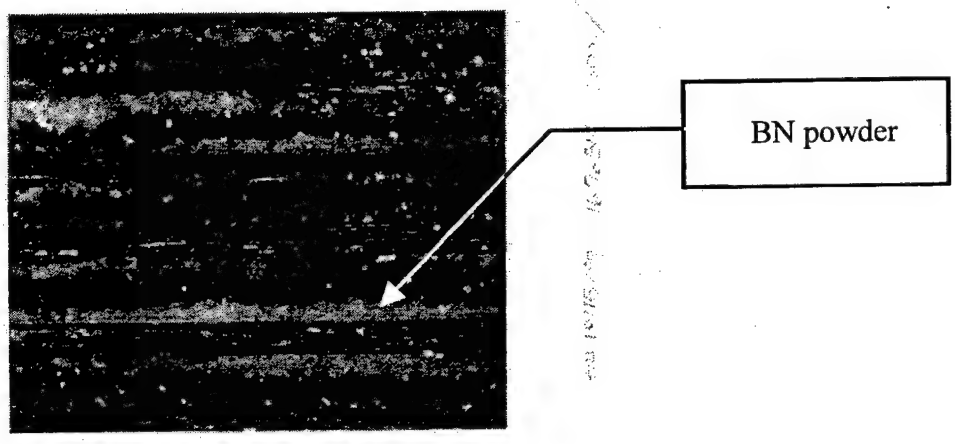

Figure 15. Sample showing 16% BN fill, viewed with polarized light

3.8.4 Discussion of results

3.8.4.1 The reason we have referred to apparent voids in Figure 12 is due to difficulty encountered while polishing the samples caused by the variation in hardness and brittleness of the matrix, carbon fiber and BN. We believe that the voids are a material property rather than being caused by the polishing due to their random distribution. If polishing caused them we would have expected a more regular signature.

3.8.4.2 The next stage of the micrograph project will be to polish the autoclave cure samples and compare the results with the press cured samples. The polishing technique will

also be further refined. If the void content is higher in the press cure samples than the autoclave samples this may explain the difference in the measured Kz.

3.8.4.3 Each of the micro-sections indicated that the BN powder remained in a layer between the plies of material and did not flow in between the fibers. This is most clearly seen when the samples were viewed under polarized light as presented in Figure 15. The variation in thickness of the BN layer is attributed to uneven distribution of the powder during lay-up, uneven flow of the powder during cure or a combination of the two.

4 Materials

Table 3. Materials used in program

Material	Supplier	Specification
Fiber	Amoco	K-800 X
Prepreg	Hexcel	K-800X 2K/954 R/C: 33% (by weight) FAW: 8365 G/M^2 Fiber Volume 61%
Boron Nitride	Advanced Ceramics	Polar Therm 620

4.1 Material selection

4.1.1 The choice of K-800X fiber was based on availability and cost rather than any technical reason. Since Kz is mainly dependent on the matrix it is believed that the results presented will also apply to laminates fabricated from other fibers.

4.1.2 Hexcel 954-3 was chosen since it is a space qualified resin and one of the applications of this technology is to fabricate thermal management components for spacecraft.

4.1.3 Boron Nitride was selected as the loading material to enhance Kz for the following reasons

- It is inert and will not cause corrosion
- Compatibility of coefficient of thermal expansion
- Commercial availability
- A literature survey indicated BN powder significantly improved the thermal conductivity of compression molding compounds.
- Density and structure of BN is similar to carbon.

5 Fabrication method

The laminates were fabricated by simply adding a predetermined weight of BN powder to plies of prepreg during a normal lay-up process. The laminates were then cured using the Hexcel recommended cure cycle for either press or autoclave.

5.1 Selection of fabrication method

5.1.1 The most important reasons for selecting this method of fabrication in preference to mixing the BN with the resin prior to prepregging is because:

- It is possible to load only specific areas of laminate subject to high thermal inputs. (This is particularly important for high performance, structural applications.), and
- It is very simple to carry out, particularly in small batch fabrication, and is lower cost than having the "prepregger" blend the powder during prepreg operation.

5.2 Details of fabrication method

Table 4. weight of BN added to laminate

% of BN added to laminate	Weight grams/in^2
0%	0.00000
2%	0.00258
4%	0.00516
6%	0.00774
8%	0.01032
10%	0.01290
12%	0.01548
14%	0.01806
16%	0.02064

The area of each ply was calculated and the weight of BN to be added was simply calculated by multiplying the area by the weight given in Table 4.

5.2.1 A detailed description of the fabrication process is presented in US patent 5,962,348, "Method of making thermal core material and material so made", authors John Bootle and Frank Burzesi and assigned to XC Associates.

6 Future Work

6.1

6.2 **Identify programs**

XCA have identified a number of programs that are interested in enhanced thermal conductivity, composite laminates. We are working with several companies to identify programs that have particular requirements that will lead to commercialization.

6.3 **Computer Model**

Develop a computer model of laminates to predict the thermal and mechanical behavior. This will be a very useful tool to use as a method to analysis candidate laminates to determine the critical factors influencing thermal conductivity. Prior work by XCA and others has demonstrated the Kz is influenced by a large number of variables. It would be impractical to build physical laminates and test each candidate and the use of a computer code would greatly assist with optimization.

Preliminary work indicates that the computer code would be written as a module for use with an established finite element code such as COSMOS/M

6.4 **Plating**

XCA have identified plating as a significant issue for composite components. In prior work, XCA has successfully plated composite thermal cores using NiCad/Chromate with excellent adhesion that resisted 500-hour salt spray testing. However, this testing required considerable surface preparation that resulted in a 64 surface finish. XCA believes that it is possible to modify the surface treatment using a corona discharge that will result in a surface better than 32.

6.5 **Production method**

Once programs have been identified it will be important to develop an automated method of production that will spread the required weight of BN powder in the correct areas of the laminate.

7 Commercialization

7.1 **Commercial applications**

7.1.1 XCA plans to market the BN filled composite laminates under the name of CHS-600, CHS-800 and CHS-1100 where CHS stands for **C**omposite **H**eat**S**inks and the number refers to the nominal value of the fiber thermal conductivity.

7.1.2 The use of high thermal conductivity, composite laminates has commercial applications where higher thermal conductivity and reduced weight are required.

7.1.3 This program has demonstrated that the addition of BN powder to a carbon fiber laminate increases the Kz. The graph shown in Figure 6 shows that a filled composite core is more efficient than an unfilled composite thermal core and will run at a lower temperature which is a major benefit for electronic applications. XCA is working with a number of prime contractors to provide material samples for evaluation for use in future programs.

7.1.4 As part of this SBIR program XCA has fabricated material samples for evaluation by Lockheed Martin, Raytheon and Johns Hopkins University Applied Physics Laboratory.

7.1.5 A survey indicates that the technology developed in this SBIR has specific advantages over other materials used for thermal applications. Table 5 presents a comparison of BN filled carbon fiber composite materials with typical materials used for thermal applications.

Table 5. Comparison of material properties

Material	Advantage of BN filled composite	Comment
Aluminum	• Higher thermal conductivity than aluminum • Thin cross-section, typically 0.060" or less • Lighter weight than aluminum • Higher stiffness and strength than aluminum • Low coefficient of thermal expansion	Aluminum is usually the baseline material of choice based on cost, but when high thermal performance or reduced weight is required composite offers definite advantages.
Exotic Metals Al.Be, Be.Be oxide, AlSi	• Similar performance • Lower cost by molding to finished shape • No health hazards associated with machining	These metals have advantages compared to composite for applications requiring complex shapes that cannot be molded.
Unfilled carbon fiber composite	• Addition of BN increases the Kz without increasing the cost	
TC1050	• Greater thermal conductivity for components less than 0.10" thick • Higher performance for thin cores • Higher stiffness	The mechanical properties of TC1050 are determined by the skin material, which means that the material becomes inefficient for thin components.

7.2 Phase II

XCA is developing a Phase II proposal with the support of a number of commercial companies who are interested in using this technology for new thermal products.

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